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# Polyimide Aerogels and Porous Membranes for Ultrasonic Impedance Matching to Air

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## Abstract

This work investigates acoustic impedance matching materials for coupling 200 kHz ultrasonic signals from air to materials with similar acoustic properties to that of water, flesh, rubber and plastics. Porous filter membranes as well as a new class of cross-linked polyimide aerogels are evaluated. The results indicate that a single impedance matching layer consisting of these new aerogel materials will recover nearly half of the loss in the incident-to-transmitted ultrasound intensity associated with an air/water, air/flesh or air/gelatin boundary. Furthermore, the experimental results are obtained where other uncertainties of the "real world" are present such that the observed impedance matching gains are representative of real-world applications. Performance of the matching layer devices is assessed using the idealized 3-layer model of infinite half spaces, yet the experiments conducted use a finite gelatin block as the destination medium.

#### 1 Introduction

Application of ultrasonic technology has developed in a wide range of fields including medicine, material non-destructive evaluation, food technology and chemical engineering, to name a few. In many applications, movement of ultrasonic energy can be accomplished by placing an ultrasonic transducer directly on the target medium. However, other applications of ultrasonic systems do not permit direct contact and the ultrasonic energy must travel through an air gap of some extent. Therefore, efficient movement of ultrasonic energy between air and other materials such as flesh, rubber or plastic, is a topic of considerable interest. For non-contact ultrasonic applications, matching the acoustic impedance of the ultrasonic transducers to air or matching the acoustic impedance of air to the target medium requires the use of an impedance matching layer to overcome transmission losses at both the air/target boundary and the transducer/air boundary. The impedance ratio can be 4000:1 for the boundary between air and flesh and 7500:1 for the boundary between a piezoelectric transducer and air. The loss in acoustic intensity associated with crossing the air/flesh and transducer/air boundaries (i.e. transmission loss) is approximately 30 dB and 33 dB respectively for a total loss of 63 dB. By comparison, direct-contact of piezoelectric ultrasonic transducers to target medium such as flesh, rubber or plastic result in impedance mismatch ratios of approximately 30:1 and a loss of acoustic intensity associated with the boundary crossing of approximately 9 dB. Direct contact of the transducer to a flesh-like target medium is, therefore, the preferred approach when such direct contact is permitted. In particular the use of ultrasonic waves for medical imaging have relied on configurations where the ultrasonic transducer is in direct contact with the skin. Yet the use of non-contact air-coupled ultrasonic waves are common in a number of disciplines. Various applications include, for example, non destructive testing and metrology, object or motion detection and distance measurement. Emerging techniques in medical applications propose using the rapeutic ultrasound on the order of 40 kHz for non-contact treatment of wounds [1]. Research has also been performed on using ultrasonics for power transmission to human implanted biochips [2], which may require frequencies above 110 kHz [3]. The use of non-contact configurations is likely to be preferred in these cases.

A number of strategies exist to maximize the total transmission power, though the most commonly used is a quarter-wavelength  $\lambda/4$  thick matching layer. The success of the design depends greatly on available materials with the required characteristic acoustic impedance and thickness in order to be used as the matching layer. Impedance matching layers for air interface applications need to have densities and sound speeds close to that of air while, for most applications, the layer must be made from a solid material. Such materials are typically uncommon or

have limited practicality. Additionally, a desired property of the matching layer material is low absorption loss [4]. The work described here investigates acoustic impedance matching materials for coupling 200 kHz pressure waves from air to materials with similar acoustic properties to that of water, flesh, rubber and plastic materials. The 200 kHz band is selected primarily due to the availability of high power transducers and associated electronics. The techniques described here apply to other ultrasonic frequencies as well, with the main difference being in matching layer thickness.

The current study determines the ability to recover transmission loss at an air/flesh boundary using a specific set of  $\lambda/4$  impedance matching layers to overcome losses associated with impedance mismatch. The impedance matching devices discussed in this work are also applicable to impedance matching of piezoelectric transducers to an air medium. Several materials are assessed for their potential to act as a matching layer. These layers include thin sheets of material that are formed from polyimide aerogels [5] and filtration membranes. The filter membranes have been characterized previously with plate/transmission methods [6]. The absorption loss, characteristic impedance and other relevant parameters have been previously determined for these filter membranes [4]. For the polyimide aerogels, only material properties such as density and Young's modulus are available. Acoustic properties such as impedance and sound speed must be derived from the material properties for the aerogel materials. In addition, attenuation coefficient values are not available for the polyimide aerogel membranes used here, a-priori. Absorption loss estimates for the aerogel materials are made as part of this work.

Assessment of the signal transmission loss recovered by the matching layer is performed experimentally using a surrogate for a flesh target medium. The effects of the impedance matching layer are analyzed in terms of a theoretical matching layer of infinite extent, joining two semiinfinite volumes of air and flesh, as commonly found in text books [7]. Other studies of ultrasonic matching materials, such as [6,8], employ extensive controls to isolate the matching layer effects. The results of these laboratory studies can be directly interpreted in the context of infinite matching layers and semi-infinite target and source volumes. Conversely, the experimental setup applied here uses a target medium of limited extent and uses a small piece of matching layer material on the target medium to act as a window for entry of ultrasonic energy. Continuous Wave (CW) sinusoids are employed as interrogation waveforms in the current experiment and no effort is made to isolate direct and reflected energy within the target medium. As a result, standing waves set up within the target medium, resulting in unmodeled cavity resonance effects. Therefore, the experimental setup provides an assessment of real-world applications that include transmission of signals with constant envelope into a finite extent medium, such as a body part or a piece of rubber or plastic. Within the context of the "real world" uncertainties associated with the experimental setup, the results described in this report provide meaningful data regarding the effectiveness of an impedance matching layer.

## 2 Methodology

#### 2.1 Quarter Wavelength Matching Layer

The characteristic acoustic impedance of a material is given by z = $\rho_0 c$ , where  $\rho_0$  is the density of the fluid and c the speed of sound in the medium. In an ideal case, total acoustic power transmission between two mediums is possible with a single quarter-wavelength thick intermediate matching layer [7]. This is achieved by selecting the intermediate matching layer impedance,  $z_2$ , to be the geometric mean of the individual material layer impedance values:  $z_2 = \sqrt{z_1 z_3}$ , where  $z_1$ ,  $z_3$  is the impedance of the source and destination materials respectively. When using  $\lambda/4$  matching techniques, typically layers are chosen with an impedance close to the ideal value of  $\sqrt{z_1z_3}$ . If layer impedance is close to the ideal value and absorption loss is low, the technique provides near perfect transmission of energy across the mismatch boundary at a single frequency. For single layer matching devices, the effect degrades quickly as the excitation frequency changes from the resonant frequency. In such cases, the matching layer provides a high level of matching gain over a narrow frequency band. Deviation from the resonant frequency therefore implies lower transmission. It is well known that multi-layered acoustic matching layers are necessary to increase the frequency bandwidth and sensitivity [9].

A single-layer quarter-wavelength impedance matching imposes a challenge for matching air to other non-gaseous mediums due the required impedance value for the matching layer. A successful design depends greatly on available low acoustic impedance materials with low attenuation and required thickness for the matching layer. Consider for example a simple case of acoustically matching the transmission from air to water. The acoustic impedance of air is on the order of 0.4 kRayl and that of water is approximately 1520 kRayl. The required impedance for a quarter-wavelength matching layer is therefore  $\sqrt{1520 \times 0.4} = 24.6 \,\mathrm{kRayl}$ . Inspection of impedance values for common materials listed in Selfridge [8] indicates that such an impedance magnitude is not common of most materials, as it is over an order of magnitude lower than solid materials and an order of magnitude or more above gases. Furthermore, for quarter-wavelength impedance matching, the required matching layer thickness imposes further restrictions on the material properties. For example, material samples with thicknesses less than a fraction of a millimeter must be sturdy for practical applications.

For evaluating the  $\lambda/4$  matching layer effectiveness, one can look at the signal transmission intensity value as altered by the addition of the matching layer material. The intensity transmission coefficient, T, is related to the intensity reflection coefficient, R, through conservation of energy such that

$$T = 1 - R. (1)$$

R is related to the complex-valued reflection coefficient,  $\mathbf{R}$ , by  $R = |\mathbf{R}|^2 = \mathbf{R}\mathbf{R}^*$ . The complex-valued reflection coefficient,  $\mathbf{R}$ , at normal incidence using a  $\lambda/4$  matching layer from medium one to medium three is given by [7]:

$$\mathbf{R} = \frac{\left(1 - \frac{z_1}{z_3}\right) \cos \tilde{k}_2 L + j\left(\frac{z_2}{z_3} - \frac{z_1}{z_2}\right) \sin \tilde{k}_2 L}{\left(1 + \frac{z_1}{z_3}\right) \cos \tilde{k}_2 L + j\left(\frac{z_2}{z_3} + \frac{z_1}{z_2}\right) \sin \tilde{k}_2 L}$$
(2)

where the sub index denotes the media layer, 1, 2, 3 and L is the matching layer material thickness. Following published techniques, such as [6], the complex wave number  $\tilde{k}$  is used to include the loss due to material acoustic absorption. The complex wave vector is defined as  $\tilde{k}=k-j\alpha=\omega/c-j\alpha$ , where k is the wave number,  $\alpha$  the longitudinal wave attenuation, c the longitudinal velocity in the medium and  $\omega$  the angular frequency.

The acoustic signal transmission properties for quarter-wavelength matching layers can be described using properties similar to a second order system at the resonance location. Figure 1 illustrates the relationship between the transmission coefficient and the transmission frequency. The resonance location,  $f_r$ , is primarily established by the matching layer thickness and the material acoustic velocity. The Q factor,  $(Q = f_r/\Delta f_r)$ where  $\Delta f$  is the half-power bandwidth) of the intensity transmission curve is strongly dependent upon absorption loss affects. In particular, the main effect of increasing absorption loss in the matching layer is a flattening of the intensity transmission response curve. The increased absorption loss reduces peak power transmission and decreases Q [4]. Complete transmission, when T = 1, is achieved only with zero absorption loss and perfect impedance matching. The filter membranes or aerogel membranes used in this work are approximately 0.5 mm thick. As such, one might expect that absorption loss would not have a strong influence on overall matching layer performance. However, the resonance phenomena occurring within the  $\lambda/4$  matching layer causes the energy to pass through the layer multiple times and therefore the absorption coefficient can have a strong influence on matching layer performance. This is especially true for cases in which the matching layer impedance is close to the ideal value [4]. Thus, the inclusion of the absorption coefficient is necessary when modeling the behavior of physical materials, regardless of thickness.

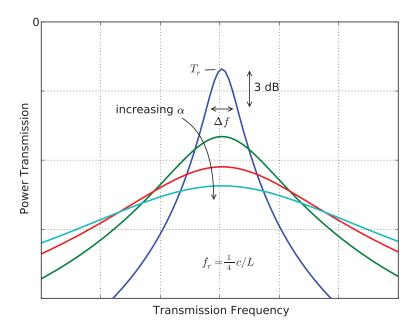


Figure 1. Illustrative relationship between the transmission coefficient and frequency as related to material absorption when all other parameters are held constant.

### 2.2 Matching Layer Materials

Porous materials are potential candidates for acoustic impedance matching as they exhibit acoustic impedance properties between that of gasses and low density solids such as plastics. Aerogels and porous membrane filters are included in this category of potential materials. Silica aerogels have been identified in the past for impedance matching layer applications [9, 10]. In practice, it is difficult to maintain a matching layer consisting of only the silica aerogel material, as the silica aerogel is easily damaged due to low density and brittle characteristics. In addition the material needs to be hydrophobic in order to withstand environmental changes and allow the use of adhesives during device manufacture. The solution has historically been to pair the silica aerogel with a porous ceramic to form the matching layer. Recent advancements in aerogel technology [5] has produced materials which are not subject to these shortcomings, producing flexible materials which are significantly less fragile and have improved moisture resistance.

For this study  $\lambda/4$  acoustic matching is performed using polyimide aerogels and porous membrane filter materials. The aerogel samples are cross-linked polyimide aerogels, where 2,2'-Dimethylbenzidine (DMBZ) or p-phenylenediamine (PPDA) is used in combination with a more flexible diamine as published in [5]. Tests were performed using the aerogels where the amount of rigid diamine was varied from 0 % to 100 % of the

| Material              |     | Density         | Young's<br>Modulus | Acoustic<br>Velocity | Impedance |
|-----------------------|-----|-----------------|--------------------|----------------------|-----------|
| [rigid diamine $\%$ ] |     | $[{ m kg/m^3}]$ | [MPa]              | [m/s]                | [kRayl]   |
| PPDA                  | 100 | 395             | 78.7               | 446                  | 176       |
| PPDA                  | 75  | 400             | 54.3               | 368                  | 147       |
| PPDA                  | 50  | 451             | 51.7               | 339                  | 153       |
| PPDA                  | 25  | 274             | 20.2               | 272                  | 74        |
| PPDA                  | 0   | 163             | 18.4               | 336                  | 55        |
| DMBZ                  | 100 | 108             | 17.9               | 407                  | 44        |
| DMBZ                  | 75  | 150             | 25.6               | 413                  | 62        |
| DMBZ                  | 50  | 197             | 21.5               | 330                  | 65        |
| DMBZ                  | 25  | 132             | 18.0               | 369                  | 49        |
| DMBZ                  | 0   | 162             | 10.4               | 253                  | 41        |

Table 1. Polyimide aerogel material properties. Material mechanical properties obtained from [5]. Acoustical impedance and velocity are calculated from theory.

total diamines in the aerogel backbone material. This work focuses on aerogels with DMBZ of 50 % and greater as these materials exhibit improved moisture resistance as indicated in [5]. In addition, as will be shown in this work, the DMBZ materials exhibit acoustic impedance values close to the necessary 24.6 kRayl value for a  $\lambda/4$  air to water matching layer.

For the aerogel samples, the acoustic properties are not readily available as acoustic property characterization has not been performed. Therefore, the acoustic properties are calculated and approximated using the average mechanical properties for density and Young's Modulus as published in [5]. In order to calculate the material acoustic properties, recall the equation for the acoustic impedance  $z = \rho_o c$ . The acoustic speed in the material is given by  $c = \sqrt{E/\rho_o}$ , where E is Young's modulus of elasticity. By substitution, the acoustic impedance as a function of solely the mechanical properties is  $z = \sqrt{\rho_o E}$ . The calculated results for the material acoustic parameters is shown in Table 1. These aerogel materials span an estimated acoustic impedance range of 41 kRayl to 176 kRayl. While none of the materials shown in Table 1 achieve the desired value of 26 kRayl, predictions of acoustic impedance for a number of the DMBZ materials indicate values that are close to ideal. As mentioned previously, this study focuses on the aerogels with DMBZ of 50% and greater due to their moisture resistance. It is important to note that the listed material acoustic impedance values also includes the necessary quarter-wavelength matching impedance for coupling typical piezoelectric transducer to air which is approximately on the order of 110 kRayl for a 30 MRayl transducer.

| Sample | Material                   | Resonant Frequency $\lambda/2$ , [Hz] | Impedance<br>[kRayl] |      |
|--------|----------------------------|---------------------------------------|----------------------|------|
|        | Pall Metricel <sup>®</sup> | 421                                   | 40                   | 820  |
| 2      | Pall Metricel®             | 412                                   | 40                   | 860  |
| 3      | FH Millipore               | 329                                   | 20                   | 1900 |
| 4      | FH Millipore               | 330                                   | 20                   | 1600 |
| 5      | Pall Supor <sup>®</sup> 2x | 476                                   | 130                  | 235  |

Table 2. Properties of porous membranes for acoustic impedance matching. Materials and characterization of acoustical properties courtesy of Dr. Gómez Álvarez-Arenas [11]. The materials were selected with the first thickness resonant frequency,  $\lambda/2$ , close to 400 kHz in order to be used as a  $\lambda/4$  matching layer at 200 kHz.

The porous membranes used in this work are commercially available filters and are not produced specifically as impedance matching materials. The samples in this study are produced by the Pall Corporation and the EMD Millipore Corporation. Specifically, for this study the Pall GN-4 Metricel® membrane filters are used, which are made of mixed cellulose esters. The FH Millipore filter membranes are made of Polytetrafluoroethylene (PTFE). In addition to the single-layer filter membranes, a double layer of the Pall Supor<sup>®</sup> filter membranes, made of polyethersulfone (PES), is also investigated. Two Pall Supor® membranes are glued together to get a measured  $\lambda/4$  resonance frequency close to 235 kHz. These filter membranes have been investigated previously as potential ultrasonic impedance matching materials [4,6]. The researcher, Tomás E. Gómez Álvarez-Arenas at the Spanish National Scientific Research Council (CSIC) graciously provided the porous membrane acoustic impedance matching samples for this study. Included with the samples are the measured acoustic material properties [11] and are found in Table 2. These acoustical properties are determined via a plate/transmission analysis (PT) [6] in which the filter membrane samples are measured with air on either side of the sample and pulses are used as interrogation signals.

It is worth noting that, while the impedance mismatch at an air and water (or flesh, plastic or rubber) boundary is not as large as the mismatch between air and piezoelectric transducers, the impedance mismatch between air and rubber-like materials (or water, flesh, plastic) is more challenging to overcome as it requires materials with acoustic impedance that is more than four times lower than that associated with an air/piezo matching layer. Indeed, examination of Table 1 indicates that the required impedance for matching air to rubber-like materials is 40 % below that of the material with the lowest characteristic impedance listed. At the same time, Table 1 indicates that the required character-

istic impedance for matching air to piezo materials lies between that of PPDA 25 and PPDA 50. Additionally, while the filter membrane materials listed in Table 2 possess acoustic impedance values that bracket the values predicted for matching air to rubber-like materials, the associated absorption coefficients are four to eight times higher than that estimated for aerogel materials (as will be shown in the results presented here, Table 4).

## 3 Experiment

The ultrasonic impedance matching test consists of passing a 200 kHz ultrasonic signal in air through the  $\lambda/4$  matching layer and into a medium with substantially different acoustic properties than that of air. For this work, a medium with acoustic properties similar to that of water, flesh, rubber and plastic materials is desired. The destination acoustic medium is represented by a gelatin block. The gelatin has similar acoustic characteristics (impedance, sound speed) to water, flesh, plastics and rubber-like materials. Even though the gelatin is a solid, a measurement sensor may be placed within the block by either molding-in during formation of the block or by placing the sensor in a water-filled cavity within the block. In addition, the use of a gelatin block allows for the matching layer samples to be easily placed easily on to the surface without excessive concern that the matching layer will absorb liquid. The experiment uses a gelatin block with a concentration of 120 g/L as the destination medium. The acoustic velocity as a function of gelatin concentration in water is found in Hall et al. [12]. The selected concentration corresponds to a speed of sound of approximately 1583 m/s. The corresponding acoustic impedance of the water-gelatin mixture is therefore  $z = \rho_0 c = 1773 \,\mathrm{kRayl}$ . For measurement simplicity, the gelatin block contains a hollow cylindrical cavity to place a measurement sensor. The cavity is filled with approximately 0.1 liter of tap water and allows a measurement hydrophone sensor to be easily placed within the medium. Furthermore, the resulting impedance mismatch between water and gelatin yields less than 0.03 dB of additional transmission loss.

#### 3.1 Experimental Setup and Procedure

The basic experiment consists of an air-coupled ultrasonic transducer, matching layer, acoustic medium and a hydrophone sensor. Refer to Figure 2 for a complete graphical depiction of the experimental setup. A Rhode and Schwartz SM300 signal generator produces the desired 200 kHz ultrasonic source. The source signal is passed through custom piezoelectric driving circuitry and then to a 200 kHz transducer from Airmar technology, model AT200. From the transducer, the signal passes through air into the  $\lambda/4$  matching layer placed on the surface of the

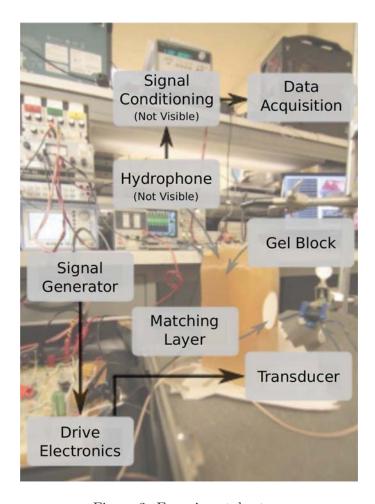


Figure 2. Experimental setup.

gelatin block. The signal is received via a Benthowave BII-7003 omnidirectional hydrophone which has a frequency range flat response between  $0.1\,\mathrm{Hz}$  to  $300\,\mathrm{kHz}$ . The hydrophone is coupled to custom signal conditioning circuitry consisting of FET-input operational-amplifiers. The resulting signal is recorded via a National Instruments 16-Bit 1.25 MS/s analog to digital converter, model NI PCI-6251. The measurements are recorded at  $1.25\,\mathrm{MS/s}$ .

The measurement procedure begins by verifying the zero-signal noise floor of the system. Using the signal generator, a sinusoidal signal near 200 kHz is focused on the gelatin block. Measurement frequencies range from 190 kHz to 218 kHz with a spacing of 2 kHz. This range of frequencies approximately coincides with the published pass band of the transducer and were selected based on the observed closed-circuit current across the transducer. The distance from the gelatin block to the piezo transducer is approximately 30 mm. At this distance, the transducer illuminates approximately a 5 mm diameter spot on the surface of the gelatin block. The transducer is positioned using a standard laboratory fixture. Once the system is initialized and the signal generator is enabled, the transducer signal is physically blocked to ensure a proper null reading on the recording device prior to the start of each experiment. The initial zero-signal reading establishes the noise floor and ensures, for example, there are no extraneous electro-magnetic interference signals from the drive electronics affecting the sensing circuitry. Next, without the addition of any matching layers, the signal on the receive hydrophone is recorded with the analog to digital converter. Approximately 15 seconds of data is taken for each excitation frequency. The recorded bare surface signal is used as a reference value during analysis to establish the increase in received signal power by adding the impedance matching layer. Next, the impedance matching material is placed onto the surface of the test object and the signal response is recorded with the analog to digital converter. The samples were found to adhere to the surface without the addition of any adhesives. The matching layer samples are approximately 50 mm square for the aerogel materials and the membrane filters are 47 mm in diameter.

#### 4 Results

The experimentally captured data are analyzed to determine the gain in sound intensity brought by the addition of the matching layer. The increase in intensity is established by comparing estimates of the Power Spectral Density (PSD) of the measured signal with the matching layer in place to the PSD of the measured signal without the matching layer. The ratio of the two power spectral densities is calculated at the excitation frequency, expressed in dB and referred to here as "matching gain".

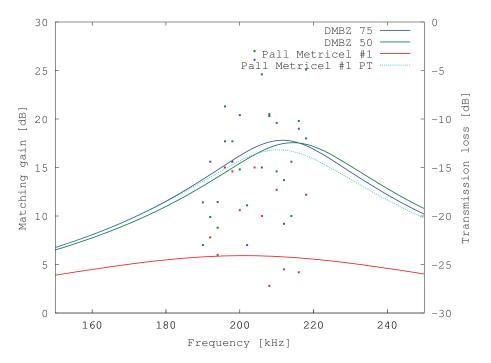


Figure 3. Acoustic energy transfer at air/flesh boundary with matching layer. Measured matching gains are depicted in points for three different matching layers. Corresponding curves for the intensity transmission coefficient are depicted with the same colors. For reference, the intensity transmission coefficient curve using parameters estimated from the plate transmission (PT) analysis is depicted for the filter membrane in dotted cyan.

| Material       |      |
|----------------|------|
| DMBZ 75        | 16.4 |
| DMBZ 50        | 16.1 |
| Pall Metricel® | 6.2  |

Table 3. Average estimated impedance matching gain over frequency, 190 kHz to 220 kHz, due to  $\lambda/4$  matching layer. Matching gain estimate is signal power observed with matching layer relative signal power observed without the  $\lambda/4$  matching layer.

In Figure 3 estimates of the matching gain derived from measured data are shown in points with blue corresponding to DMBZ 75 material, green corresponding to DMBZ 50 material and red representing the first sample of Pall Metricel<sup>®</sup> filter membrane. Representative values for matching gain provided by each of these materials are shown in Table 3. These values are obtained by averaging over the range of excitation frequencies. Note that the two aerogel materials provide an average of over 16 dB improvement in sound intensity over the band of interest. This is about 10 dB higher than that of the Pall Metrical filter membrane and represents a recovery of nearly half of the 30 dB of sound intensity "lost" at a bare air/gelatin boundary.

In addition to the vertical scale indicating matching gain on the left of Figure 3, a vertical scale indicating an equivalent intensity transmission value is on the right side of the graphic. Matching gain is related to the intensity transmission coefficient through the nominal transmission loss of sound intensity associated with an air/gelatin boundary of approximately 30 dB, (i.e. matching gain is approximately equal to transmission intensity plus 30 dB). The intensity transmission scale provides an alternate interpretation of the matching gain values. In addition to the matching gain estimates for the three different materials, Figure 3 contains intensity transmission coefficient curves, Equation 1, for each of the three materials investigated. These curves are colored to indicate the materials they are associated with, as previously described. An intensity transmission curve for the Pall Metricel® material using acoustic parameters determined by caliper measurements and plate/transmission analysis is provided for reference purposes. This curve is depicted as a dotted, cyan curve in Figure 3.

The three curves in Figure 3 are obtained by performing a least-squares fit of empirical matching gain data to Equation 1. In this fitting process, the attenuation coefficient,  $\alpha$ , and layer thickness, L, are varied so as to minimize the mean squared fit error between theoretical values of T and the measured values at each excitation frequency. The absorption loss and membrane thickness are thereby estimated for the given matching layer device from the measured intensity transmission

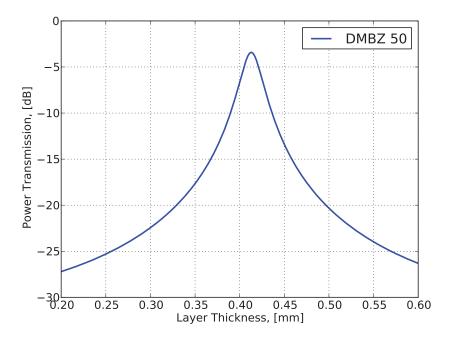


Figure 4. Theoretical power transmission for DMBZ 50 polyimide aerogel sample at 200 kHz as a function of matching layer thickness. The curve assumes an ideal material with no absorption present.

loss. The absorption loss and thickness parameters are the chosen parameters of interest for a number of reasons; 1) absorption loss data is not available for the aerogel materials 2) sample material thickness was not tightly regulated 3) thickness and absorption loss were thought to be most sensitive to sample handling and measurement errors 4) the small spot size that the transducer places on the matching layer will emphasize small variations in sample thickness that exist over the extent of the layer.

The power transmission gain is dependent on material thickness, as indicated by the relationship in Equation 1 and Equation 2. For waves incident on the matching layer at the resonant frequency, the intensity transmission will increase as the layer thickness approaches the resonance condition. Resonance in the matching layer occurs when the thickness of the material is equal to a quarter wavelength (i.e.  $L = c/4f_r$  where c is the sound speed in the matching layer and L is the layer thickness and  $f_r$  is the resonant frequency). The effect of varying layer thickness on transmission loss is illustrated in Figure 4 for the case in which a 200 kHz wave is incident on a quarter wavelength matching layer with an impedance of 65 kRayl (i.e. the nominal impedance used for the DMBZ 50 material). Note that a 0.05 mm change in thickness can produce a change in the intensity transmission of 10 dB.

| Material       | Attenuation       |                   | Thickness |       | Resonance        |       |
|----------------|-------------------|-------------------|-----------|-------|------------------|-------|
| Material       | (PT)              | (fit)             | (caliper) | (fit) | (nom)            | (fit) |
|                | $[\mathrm{Np/m}]$ | $[\mathrm{Np/m}]$ | [mm]      | [mm]  | $[\mathrm{kHz}]$ | [kHz] |
| DMBZ 75        | NA                | 246               | 0.51      | 0.49  | 203              | 212   |
| DMBZ 50        | NA                | 320               | 0.40      | 0.39  | 206              | 214   |
| Pall Metricel® | 820               | 2728              | 0.17      | 0.18  | 211              | 201   |

Table 4. Material parameters for polyimide aerogel and membrane filter matching layers. Matching layer parameters are determined by plate/transmission (PT) analysis or by fitting measured data to transmission loss curves.

Modeling the measured data with an intensity transmission curve enables an understanding of how matching layers could be modified to produce additional matching gain. As the location of the matching layer resonance frequency may not coincide with transducer pass band (i.e. where the data is), fitting an intensity transmission curve to the data may indicate a resonant peak for the layer that is outside the passband of the transducer. In such a case, the resonant peak of the matching layer and the passband of the transducer may be brought together. For example the resonant frequency of the matching layer can be altered by simply changing the thickness of the layer. The net result is an improvement in the overall matching gain.

Nominal and modeled matching layer parameters for the aerogel and membrane filter matching layers are summarized in Table 4. Absorption, thickness and the associated resonance frequency parameters for the three materials are listed as provided via plate/transmission analysis as well as those obtained by the curve fitting process. The curve fits for the two polyimide aerogel materials (Figure 3) show similar resonance peak locations and absorption loss numbers. Curve fits for all three materials show broad resonance (relative to transducer passband). Absorption loss numbers for the polyimide aerogels are low compared to that estimated for the filter membranes. The estimated low loss values are consistent with the significantly larger measured matching gain numbers associated with the aerogel layers. Resonance locations of the modeled intensity transmission values do not extend beyond the data collection interval and thus the modeling process does not predict large matching gains through suitable modification of the layers.

## 5 Analysis and Discussion

The measured power transmission gain at a single frequency is consistent between multiple measurement runs. Variations in the observed power transmission gain are primarily a result of: 1) layer adhesion, 2) in-

cident angle. In this experiment, the matching layer materials adhere to the gelatin block without the addition of adhesives. Still, care must be taken to ensure a consistent adhesion of the material, as any trapped air will increase the absorption coefficient. In addition, this experimental setup did not tightly control the stand-off distance of the transducer and the angle of incidence relative to the matching layer surface normal. The geometry of the transducer and gelatin block were set primarily by eye. As a result, the effects of matching layer adhesion to the block, variations of ultrasonic wave angle incidence on the block and location of the beam placement on the block are not controlled with exacting precision.

It is also worth noting that the porous nature of the materials used in this experiment makes them subject to some water absorption. Based on observed minor material color distortions, it is clear that the materials absorb some water and residual vegetable oil<sup>1</sup> out of the gelatin block. Although no variation in the measured power transmission gain was observed over the course of the measurements, the addition of water in the material porous structure will alter the material acoustic properties.

The thickness of the polyimide aerogel thin films used in this study are not tightly regulated during the manufacture process. The material does exhibit some shrinkage during processing. The polyimide aerogel samples are produced with a nominal thickness of 0.5 mm, yet the resulting thickness will range from 0.3 mm to 0.6 mm. Furthermore, the samples were not altered or pre-processed to polish the surface. The samples were used in this study as received without alteration. As a result, the material thickness is not uniform over the sample, nor is the material completely flat as currently produced. The measured material thickness variation is on the order of 0.05 mm or more depending on the sample. As shown in Figure 4, the power transmission gain is strongly dependent on the material thickness. This work used a fit to the material thickness, resulting in an "effective" or average thickness over the area exposed by the ultrasonic signal. Future work will likely include some pre-processing or polishing of the polyimide material to ensure consistent geometric properties.

Observation of the results in Table 4 shows fit material thickness values for the aerogel materials less than the caliper measured value. It should be noted, that the actual material thickness may also be affected by the nature in which the experiment was conducted. Over the course of the experimental measurements, all the samples were re-used for repeated measurements at various configurations. As part of the experiment setup, the matching layer would be rubbed slightly to the gelatin block in an attempt to remove trapped air and to ensure good adhesion. After repeated use, the samples could become slightly "compressed" from the initial state. The change in the material thickness will result in a shift of the material peak resonant frequency. Caliper

<sup>&</sup>lt;sup>1</sup>Vegetable oil was used to help release the gelatin block out of the form.

measurements of the material thickness listed in Table 4 corresponds to the value measured after the most recent experimental run, consistent with the depicted experimental data in Figure 3.

For the Pall Metricel® membrane, Table 4 indicates a fit value for the material thickness to be slightly greater than the nominal material value. The increase is likely due to measurement noise in the measured power transmission gain. In this experiment, it is expected that the aerogel materials will be more affected by material compression than the filter membranes. The filter membranes are thinner material than the aerogel samples and only required little effort to remove air bubbles during material application. As such, less force is applied when placing the filter membrane material on the gelatin block.

The materials used in this work are primarily discussed with respect to acoustic matching layer properties. It is worth discussing here a few other properties of the evaluated materials. For instance from a practical usage perspective, the thin film FH Millipore membranes are very fragile and difficult to work with. Thus, the FH Millipore membranes are not practical for use as a matching layer for an air/flesh boundary as currently readily available. As a result, this material was not heavily investigated in this study. In addition, the filter membranes in general are not currently manufactured with the goal of producing a material with consistent acoustic properties. As such, the acoustic properties are different between random samples. The filter membrane samples used in this work were previously experimentally evaluated and were selected to exhibit a peak resonance suitable for a 200 kHz  $\lambda/4$  matching layer.

In comparison, the polyimide aerogel materials are more robust to work with and handle. The material is flexible and pliable. The aerogels produced with  $100\,\%$  DMBZ are slightly more brittle than those with lower DMBZ percentages, but can still be flexed slightly without cracking. The aerogels made with at least  $50\,\%$  DMBZ are water resistant [5], whereas the PPDA are not as water resistant and after repeated or extended use, water damages the porous structure of the material and hence the acoustic properties.

Parameters determined by the plate/transmission method for the Pall Metricel<sup>®</sup> filter membrane (cyan/dotted curve in Figure 3) predict a transmission loss similar to that indicated by the aerogel layers. The absorption coefficient for the filter membrane determined via curve fitting is more than that determined by plate/transmission analysis. The discrepancy between the estimated absorption coefficient and the plate/transmission analysis is likely due to the experimental setup implemented here. Use of the finite gelatin block for the acoustic medium introduces ultrasound reflections at the boundaries of the gelatin and air and result in standing waves within the material.

The intensity transmission data are not well modeled by the curves. In particular, the statistical goodness-of-fit R-square numbers for fitting the aerogel data to the curves are very close to  $13\,\%$ –usually considered

a poor fit when statistical methods are applied. Additionally, the R-square for the filter membrane device (Pall Metricel® #1) is only 0.3%. Nonetheless, the difference between thickness measured by caliper and that determined by the curve fit is about 5% or less for all cases. Changes in cavity resonance occurring within the gelatin block brought by the introduction of the matching layer is a likely candidate for the observed variations. Within the context of the modeling process implemented (i.e. the curve fitting) such unmodeled dynamics can only be interpreted as a high level of measurement error. The errors in the intensity transmission measurements do not have a discernible frequency dependence and appear to be spread uniformly over the passband.

Recall that an increased value for the absorption term,  $\alpha$ , results in an overall flatter power transmission curve when plotted with respect to frequency. Indeed, inspection of Figure 3 depicts a measured intensity transmission curve for the Pall Metricel® sample which is less peaked or flatter across the frequency band compared to the curve calculated using a thickness parameter from caliper measurements and a characteristic impedance from the PT method. Due to the high level of measurement uncertainties, flattening of the intensity transmission curve is realized in the curve-fitting process by introducing increased levels of absorption. Therefore, results for the measured absorption parameters determined by this method are expected to be an over estimate. As a result, the actual aerogel matching intensity transmission loss values are expected to posses a stronger frequency dependence and have higher peak values than that depicted in Figure 3.

## 6 Summary

Experiments conducted here indicate that the filter membrane devices yield about 6 dB of matching gain for the boundary between air and a gelatin destination medium and recently developed DMBZ crosslinked polyimide aerogels provide about 16 dB of matching gain over a broad range of frequencies covering 200 kHz. This is important for applications in which ultrasound is to be transferred into water or flesh or plastic/rubber materials in a setting in which contact between the ultrasonic transducer and the destination medium is undesired. Further note that the PPDA cross-linked polyimide aerogels appear to have acoustic properties suitable for matching ceramic piezoelectric transducers to air. Absorption loss number for the polyimide aerogels are indicated to be low when compared to many of the filter membrane materials. Indeed, it is these materials that produced the best overall matching gain estimates. Future work should include a laboratory-grade characterization such as the plate transmission method as provided in [6] or the reflection-based methods described in [8] in order to fully understand the acoustic properties of these new materials. Such testing should also include experiments to assess the ability of the PPDA aerogels to match piezoelectric transducers to air.

The experiments were not conducted with laboratory-like controls over parameters such as relative orientation of the transducer to the gelatin block or separation of direct and reverberant signals. Ideal matching layer thickness was not obtained and adhesion of the matching layer to the gelatin block was not always perfect. The use of a finite gelatin block introduces a number of possibilities for the ultrasound to reflect within the block and for nodes and anti-nodes to form. The location of these nodes will move as the excitation frequency changes and with the introduction of the acoustic matching layer. This introduces complex variations in the matching gain measurements. While the analysis of the data is complicated by the resulting "measurement error" or unmodeled dynamics, the estimated resonance locations (as determined by the curve fitting process) are within the pass band of transducer for all layers for which data are available. The relatively informal processes of the experiment that cause the data to portray power recovery that would occur in conditions where controls on the relative orientation of the transducer a destination medium is marginal, where effects of reflection and reverberation within the article can be significant. As a result, this study indicates that these new materials can be used to form impedance matching layers in which nearly half of the loss in the incident-to-transmitted ultrasound intensity associated with an air/water, air/flesh or air/gelatin boundary can be recovered where other vagaries of the "real world" are present.

#### References

- 1. Ennis WJ, Foremann P, M. N. e. a.: Ultrasound therapy for recalcitrant diabetic foot ulcers: results of a randomized, double-blind, controlled, multicenter study. *Ostomy Wound Manage*, vol. 51, no. 8, 2005, pp. 24–39.
- 2. Hu, Y.-C.; Liao, P.-L.; Shih, W.-P.; Wang, X.-Y.; and Chang, P.-Z.: Study on the acoustic impedance matching of human tissue for power transmitting/charging system of implanted biochip. *Nano/Molecular Medicine and Engineering (NANOMED)*, 2009 IEEE International Conference on, 2009, pp. 201–205.
- 3. Jr., F. R. A.: *Nanomedicine, Basic Capabilities*, vol. 1. Landes Bioscience, 1999.
- 4. Gómez Álvarez-Arenas, T. E.: Acoustic impedance matching of piezoelectric transducers to the air. *Ultrasonics, Ferroelectrics and Frequency Control, IEEE Transactions on*, vol. 51, no. 5, 2004, pp. 624–633.

- 5. Guo, H.; Meador, M. A. B.; McCorkle, L.; Quade, D. J.; Guo, J.; Hamilton, B.; and Cakmak, M.: Tailoring Properties of Cross-Linked Polyimide Aerogels for Better Moisture Resistance, Flexibility, and Strength. *ACS Applied Materials & Interfaces*, vol. 4, no. 10, 2012, pp. 5422–5429. URL http://pubs.acs.org/doi/abs/10.1021/am301347a.
- Gómez Álvarez-Arenas, T. E.: A nondestructive integrity test for membrane filters based on air-coupled ultrasonic spectroscopy. *Ultrasonics, Ferroelectrics and Frequency Control, IEEE Transactions* on, vol. 50, no. 6, 2003, pp. 676–685.
- 7. Kinsler, L.: Fundamentals of Acoustics. Wiley, 4th ed., 2000.
- 8. Selfridge, A. R.: Approximate material properties in isotropic materials. *Sonics and Ultrasonics, IEEE Transactions on*, vol. 32, no. 3, 1985, pp. 381–394.
- 9. Aegerter, M.; Leventis, N.; and Koebel, M.: Aerogels handbook. Advances in sol-gel derived materials and technologies, Springer Science+Business Media, LLC, 2011. URL http://books.google.com/books?id=3FXWzJFOlnAC.
- 10. Gomez Álvarez-Arenas, T. E.; Montero de Espinosa, F.; Moner-Girona, M.; Roig, A.; Molins, E.; et al.: Viscoelasticity of silica aerogels at ultrasonic frequencies. *Applied physics letters*, vol. 81, no. 7, 2002, pp. 1198–1200.
- 11. Gómez Álvarez-Arenas, T. E.: Personal correspondence, July 2012.
- 12. Hall, T.; Bilgen, M.; Insana, M.; and Krouskop, T.: Phantom materials for elastography. *Ultrasonics, Ferroelectrics and Frequency Control, IEEE Transactions on*, vol. 44, no. 6, 1997, pp. 1355–1365.